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PHOTOCONDUCTIVITY OF HIGH-VOLTAGE SPACE INSULATING MATERIALS

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SUMMARY

The dark and photoconductivities of four high-voltage spacecraft insulators, Kapton-H, FEP Teflon, Parylene, and fused quartz, were studied under a variety of conditions intended to simulate a space environment. All measurements were made in a vacuum of less than 10^{-5} torr while the temperature was varied from 22° C to 100° C. Some of the samples used employed conventional deposited metal electrodes--others employed electrodes composed either of an electron beam or a plasma formed by ionization of the residual gas in the test chamber.

Kapton, a technically important material in satellite construction, was found to have many unusual conduction properties. Its conductivity decreased by more than an order of magnitude when heated at 100°C in a vacuum, but ultimately attained a stable and reproducible value. The photoconductivity of Kapton-H under 2.3 suns of xenon illumination was as great as 10⁵ times the initial dark conductivity. The dark conductivity after illumination was as much as three orders of magnitude greater than the dark conductivity prior to illumination, indicating possible permanent changes in the material.

Both Teflon and fused quartz had high dark resistivities but low photoresistivities when exposed to UV. The high photocurrents are thought to arise from photoemission of electrons from the negative gold electrode upon exposure to UV in the 200-to-300-nm band. Blocking this part of the spectrum reduced the photocurrent by factors of 10 to 20. Optical-density measurements revealed that both materials transmitted UV with little attenuation.

Parylene was found to have a low but relatively stable resistivity—comparatively minor changes occurred upon heating or illuminating the sample. Optical-density measurements showed that Parylene was absorbent in the UV and would prevent photoemission from the metal electrode on the back surface.

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I INTRODUCTION

It is sometimes advantageous to design a spacecraft solar panel to generate the power at a high voltage. This permits the direct operation of some devices at the panel voltage without a dc-to-dc converter, and also reduces the i²r losses and thereby improves the efficiency of the system. Generally, systems dissipating the least heat are also the most compact and lightweight.

The electrical inculators used in such a solar panel must perform reliably and predictably in the space environment. The characteristics of this environment differ so significantly from those usually experienced in terrestrial applications, however, that the designer must have specific information on the behavior of each insulator under a wide variety of conditions. Since the spacecraft is surrounded by a conducting plasma, the reliability of the insulation is more important than would be the case if the plasma were absent. Three characteristics of the space environment that can be expected to have a significant influence on the insulator's properties are the vacuum, the temperature, and the presence of unfiltered solar radiation.

This program was undertaken to generate experimental data on the conductivity of candidate high-voltage insulating materials under the conditions listed above, and to correlate these data with available theoretical analyses. It is anticipated that the results of this work will assist designers in selecting materials and in predicting changes in the electrical conductivity of these insulators in space.

The materials studied in this program were: FEP Teflon, Kapton-H, Parylene, and fused quartz. Teflon is a well known and widely used

insulating material, and was selected for study due to its good insulating properties in terrestrial applications. Kapton, a polyimide film, is not as well known as Teflon but has properties that make it preferable to Teflon in many applications. For example, it has good mechanical strength at temperatures as high as 500°C and is more easily bonded to other materials than is Teflon. It retains its mechanical stability after prolonged UV radiation, and is being used with increasing frequency in thermal-control blankets, in solar panels, and in other space applications. These desirable characteristics and its frequent usage suggested that this material should also be evaluated.

Fused quartz is used as cover material for solar cells and for backsurface mirrors for thermal control on some spacecraft. Unusual dielectric breakdown phenomena, possibly leading to RF pulses that interfere
with digital equipment, have been observed in silvered fused quartz
samples in our laboratories. This usage, and the possibility of electrical interference caused by dielectric breakdown, led to the selection
of quartz as a material for study.

The use of Parylene coatings for insulation is relatively new, but its unique physical properties make it of interest for many future space-craft applications. The unique feature of Parylene is that it is deposited at subatmospheric pressures and polymerizes upon contact with the substrate. This leads to unusually pin-hole-free coverage on samples having complex geometries—for example, on circuit boards. Its electrical properties were also measured.

All the measurements reported here were made in a vacuum of 10^{-5} torr or less to simulate a space environment.

Measurements were made on samples in two basic configurations and in two basic environments. First, samples of each of the materials listed above were prepared with gold electrodes on opposite surfaces of

the samples. (In the case of Parylene, one of the electrodes was the copper on which the Parylene was polymerized.) Direct electrical contact was made to both electrodes. Second, samples of Kapton were prepared with metal electrodes on only one surface. Direct electrical contact was made to the metal electrode on these samples. The electrode on the first surface of these samples was comprised of either an electron gas from an electron gun, or a plasma of the residual gas in the vacuum chamber. In the case of the plasma electrode charged particles of either polarity could be withdrawn from the plasma. The purpose of these two types of measurements was to distinguish between features of the conduction process that were characteristic of the electrodes, and features that were intrinsic to the sample material itself.

Measurements were made of the bulk and the surface conductivity of these samples both in the dark and with various intensities of xenon lamp illumination. The intensity of the illumination was varied as was the spectral content of the light. The latter variation was performed with a series of band-pass filters.

In addition, measurements of the conductivity were made at temperatures of 22, 66, and 100°C while each of the above parameters was varied. Three thicknesses of Kapton and Teflon were studied. Quartz and Parylene were investigated with only one sample thickness.

It will be readily appreciated that a great number of measurements were made during this study. Furthermore, the variability of results from sample to sample required that many experiments be repeated to achieve an acceptable level of confidence in the results. This was particularly true of measurements on Kapton for which measurement procedures had to be established to achieve reproducible results.

No attempt was made to compile an exhaustive catalog of properties of these materials under all possible space environmental conditions.

Rather, an effort was made to test each material under a wide variety of conditions, concentrating on any behavior that deviated significantly from what might be regarded as "usual behavior." In this respect, phenomena that might be unusually beneficial or unusually detrimental in high-voltage applications on spacecraft were singled out for special cortion. Therefore, results on different topics addressed in this rewere not studied in the same depth, but are those thought to be of greatest importance to the spacecraft power-system declarate.

It will be shown later that a radical photoconductive behavior of Kapton was observed in these experiments. This result is of particular significance in that Kapton is being widely used in spacecraft and is replacing other insulators in many cases. The sharply increased conductivity of Kapton might be useful in solving a problem whose magnitude is only now becoming apparent—the problem of spacecraft charging.

II APPARATUS, SAMPLES, AND PROCEDURES

A. Apparatus

The apparatus used in these experiments is pictured in Figure 1. Samples were mounted in the vacuum chamber shown in the center of the photograph, and could be inspected through the glass port. The xenon lamp, neutral density and bandpass filters, and optical shutter are located in the black-painted enclosure located to the left of the vacuum chamber. Appropriately filtered light was transmitted into the chamber through a 6-inch-diameter fused quartz window in the chamber wall. Bulk currents, conducted through the samples, were measured with a Hewlett-Packard 425A picoammeter located on the optical enclosure. This instrument, like many other parts of the apparatus, was operated at elevated potentials and was enclosed in a styrofoam and plexiglas case to protect personnel and avoid stray currents. An isolation transformer was used to avoid stray currents or dielectric breakdown in the power line.

A second HP 425A picoammeter was used to measure surface currents. In this case, currents flowed between the center electrode on the sample and a concentric guard ring (to be described later). The picoammeter was connected between the guard ring and ground. Since the guard ring, for this measurement, was at ground potential, an isolation transformer was not required for this meter.

A highly regulated (0.025%), 20-kV power supply (Power Designs Pacific HV 1584 R) was used to apply potentials to the samples. Voltages were measured using a Tektronix P6015 high-voltage probe in conjunction with a HP 412A voltmeter. This power supply and voltmeter are located on the table in the left-hand portion of Figure 1. The xenon lamp used

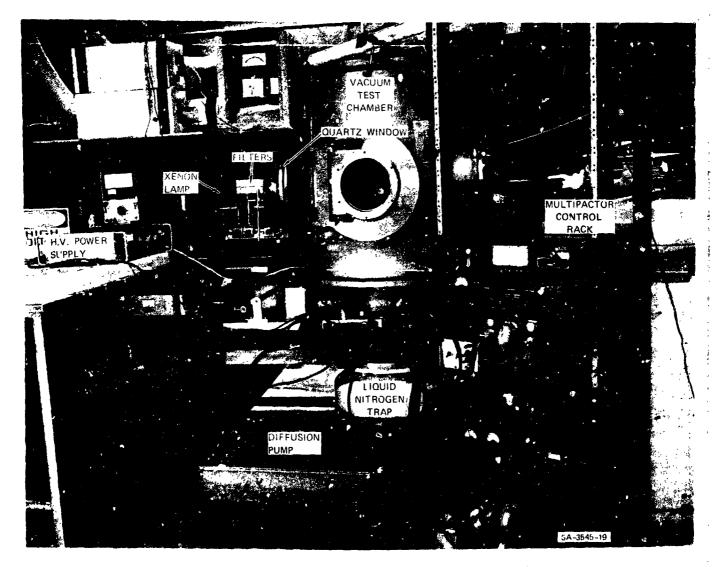


FIGURE 1 APPARATUS USED FOR TESTING INSULATORS IN A SIMULATED SPACE ENVIRONMENT

was a Varian Associates Model VIX 1500 UV with an aluminum reflector. d supphire window to enhance the output in the ultraviolet and IR portions of the spectrum.

Experiments performed with free-charge carriers as one of the electrodes used either an electron gun or a multipactor discharge to produce the charge carriers. The electron gun (upper left part of Figure 2) was of a more or less conventional design, and could be operated at elevated

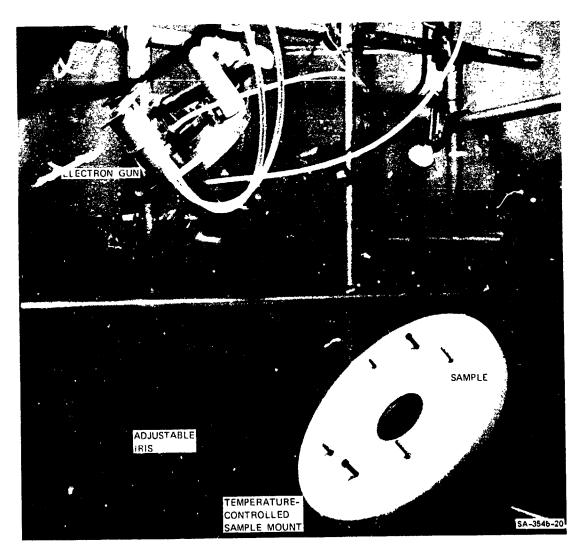


FIGURE 2 SAMPLE HOLDER (lower right) AND ELECTRON GUN (upper left).

Adjustable iris (lower left) limits area of sample exposed to xenon light.

potentials. The electron beam could be focused to concentrate it on the center section of the sample where the guarded electrode was located.

Plasmas were produced using an RF-powered, parallel-plate multipactor discharge. In this device, electrons are accelerated and collide with the plates. The electrons multiply with successive collisions because the secondary emission coefficient of the plates is greater than one. Gas molecules in the gap between the plates are ionized by collisions with the electrons. Both electrons and ions diffuse out of the plate area and form the free-charge-carrier electrode. The transmitter used with the multipactor can be seen in the rack in the right-hand side of Figure 1. The power supply for the electron gun is not shown.

Two types of sample holders were used in these experiments, depending on whether a metal or a free-charge electrode was used. In both cases provisions were made for guarding the electrode used in the actual measurement to avoid current leakage to the measurement electrode.

The sample holder used in the free-charge carrier experiments is depicted schematically in Figure 3 and can be seen, without a sample installed, in Figure 2. The assembly consists of the electrodes, 1 Teflon pressure plate used to hold samples in contact with the electrodes, and the thermal control assembly. The sample holder used in the metal-electrode experiments is shown schematically in Figure 4.

Two separate concentric metal rings can be seen in the font surface of the sample holder used in the free charge carrier experiments. The center electrode is at the same potential as the outer annulus and is guarded by it to avoid leakage currents from the center electrode to other parts of the circuit. Only the current in the center, guarded electrodes was normally measured. Similar precautions were taken on the back side of these two electrodes. Buried in the epoxy beneath the two visible electrodes is an additional electrode having an outer diameter equal to that of the annular guard ring. This electrode is electrically connected to the guard ring.

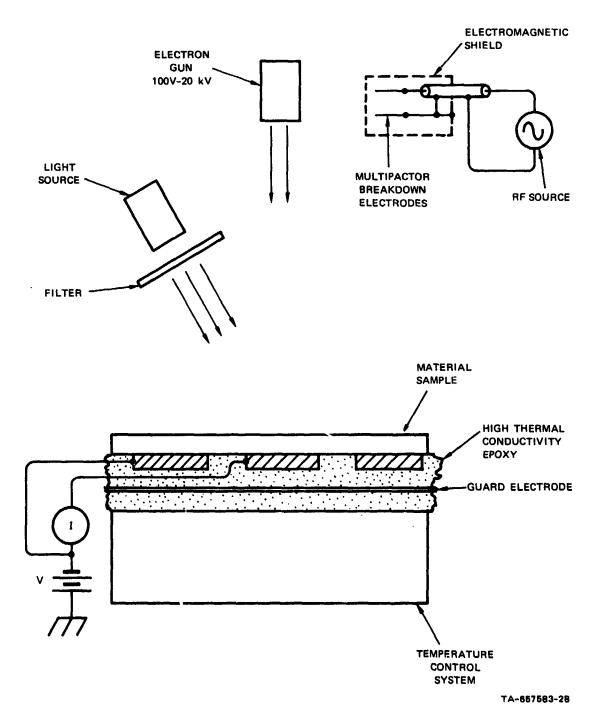


FIGURE 3 SCHEMATIC OF TEST SETUP OF EXPERIMENTS USING PLASMA ELECTRODES

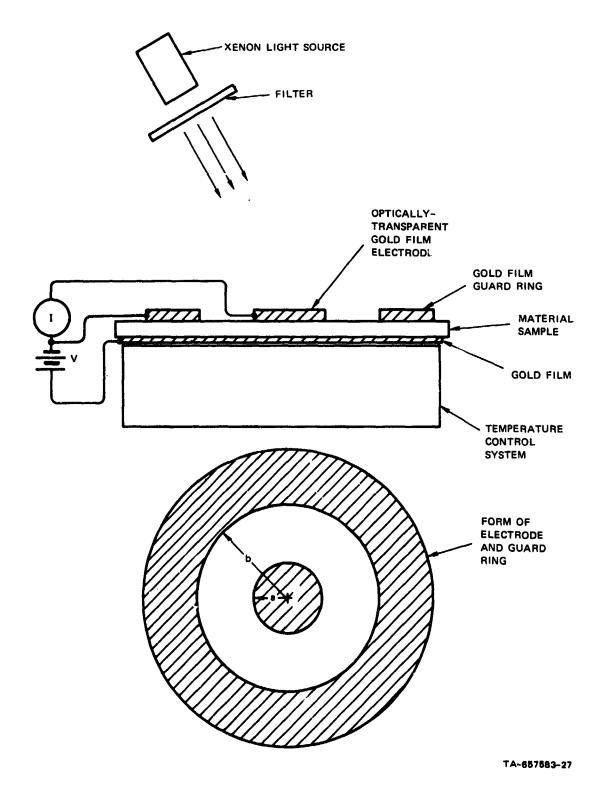


FIGURE 4 SCHEMATIC OF TEST SETUP FOR EXPERIMENTS INVOLVING DEPOSITED ELECTRODES

The epoxy used in constructing the sample holder (Emerson and Cuming stycast 2850 kt) is loaded with a high-thermal-conductivity material (note granular character of epoxy between electrodes in Figure 2). The sample holder contained an electrical heater and copper coils through which water or air could be passed for cooling. The temperature was measured by thermistors located between the Teflon plate and the epoxy block. Typically, the temperature was controlled to $\pm 1^{\circ}$ C during the experiments.

When measurements were made on samples with metal electrodes on both surfaces the epoxy sample mount discussed above was removed and the samples were placed directly on the temperature-control assembly. Contact was made to the annular gold guard ring deposited on the first surface of the sample by means of a brass ring pressed against it. Similarly, contact was made with the guarded electrode by a small brass rod.

B. Samples

Samples of Kapton-H and quartz were cleaned with isopropyl alcohol, lightly brushed, and dried in clean air prior to the sputtering of gold electrodes onto the surfaces of the samples. FEP Teflon was treated in the same manner, but was not brushed due to the resultant surface abrasion. Parylene samples were deposited on polished copper plates by the Hughes Corporation and were tested as received.

All samples were 0.1 m (4-inches) square or round. The optically transparent front-surface gold electrodes were nominally 20 nm (200 Å) thick; the back-surface gold electrodes were opaque. Electrodes were deposited on the front surface as follows. A central circular electrode 0.0302 m in diameter was deposited and served as a guarded electrode for all bulk-conductivity measurements. A second annular electrode having an inside diameter of 0.0332 m and an outer diameter of 0.076 m was used as the guard electrode. The 0.0015-m gap between the two electrodes was used for surface-conductivity measurements.

C. Procedures

It is well known that the current through a dielectric can vary appreciably with time after the voltage is applied to the sample. For this reason, it is conventional to record the current at a specified time after the voltage is applied. Unless noted otherwise, all current measurements reported here were made after the voltage was applied for 60 s. The capacitance of the sample configured as discussed above is expected to be in the range of 10^{-9} to 10^{-10} farad, and, in conjunction with the 10^7 -ohm protection resistor, results in a charging time constant of 10^{-2} to 10^{-3} s. Consequently, one would expect the current to be virtually independent of time after 60 s. It was occasionally noted that the current continued to change rapidly after 60 s. This effect is discussed in a later section.

The measurement of photocurrents entailed the specification of two times--the duration of the voltage and the duration of the light. There was a reluctance to turn the light on immediately after the voltage was applied, since the current became excessive from the combined effects and sometimes led to a breakdown of the sample. For this reason, the sample was exposed to the light after the voltage was applied for 30 s, and the current was measured 30 s later.

The occurrence of dielectric breakdown of a sample sometimes resulted in the loss of a considerable amount of time and data, so measurements were made over a conservative voltage range when possible. What constituted a conservative voltage was not known initially, and many samples were consequently damaged.

It will be shown later that apparently permanent changes were caused in Kapton by exposing it to xenon light. After this was discovered, a procedure was followed in which dark bulk and surface conductivity measurements were made at all temperatures prior to exposing the sample to

light. As a precaution, the same procedure was usually followed with the other materials, although no permanent changes were observed to be induced by the light.

III EXPERIMENTAL RESULTS

The results of the experiments performed in this program are summarized in this section. The results of measurements on each material studied are compiled separately to facilitate reference to the properties of each material. Comparisons between materials and further discussions will be found in Section IV.

A. Kapton

The experiments performed on Kapton revealed several unexpected characteristics that either were not observed in other materials or were present to a significantly lesser extent. Since the study of this material revealed possible effects that might occur in other materials, the results of this study are discussed first.

1. Dark Conductivity, Bulk

The dark, bulk conductivity of Kapton is a function of many parameters and of the time history of these parameters. The following characteristics of the dark, bulk conductivity of Kapton have been observed:

- (1) It decreases with time after being placed in a vacuum.
- (2) It decreases with time after an electric field is applied.
- (3) It initially increases and then decreases with time while being heated in a vacuum.

- (4) It approaches an asymptotic limit after Items 1,2, and 3 are repeated a sufficient number of times;further changes are negligible.
- (5) It is strongly affected by exposure to xenon light.

In addition to the above, Kapton is polarized as a result of the application of an electric field and becomes a source of emf. The current resulting from this emf is hysteretic in nature and can be reduced in magnitude by cycling the polarity and magnitude of the voltage.

The dark, bulk currents measured during the initial test at 22°C of six samples of three thicknesses of Kapton are shown in Figure 5. Two features of this figure are noteworthy. First, samples of the same thickness had nearly equal bulk currents, and second, the current is well approximated by a sinh function of the voltage at high fields. behavior is characteristic of ionic conduction in solids. The changes that occur in the conduction characteristics of Kapton after repeated heating, voltage application, and prolonged evacuation are illustrated in Figure 6. It is seen in this figure that the conductivity at room temperature can exceed the conductivity at 100°C under certain conditions. It is also seen that not only the magnitude of the current, but also the variation of the current with the applied voltage changes upon aging of the sample, and a sinh function no longer fits the data. This will be discussed in Section IV, but appears to be caused by the depletion of ionic charge carriers, by outgassing, and by migration to, and trapping at, the sample surface under the influence of the electrical field.

The results obtained immediately after evacuation are those that are likely to be observed soon after a satellite reaches orbital altitude. The dark, bulk current in one sample, measured as a function of time in the vacuum at 100° C, is shown in Figure 7.

A considerable effort was devoted to untangling the various effects noted above. Although the observed changes in the conductivity were

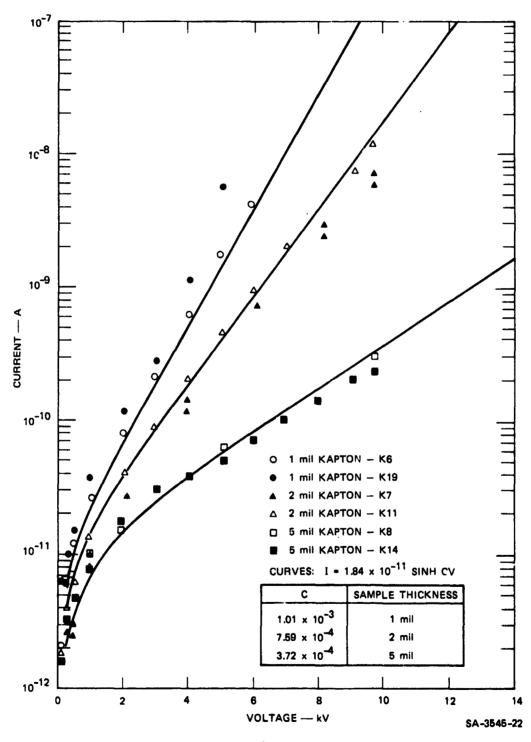


FIGURE 5 DARK, BULK CURRENT AT 22°C IN KAPTON SAMPLES BEFORE HEATING IN VACUUM

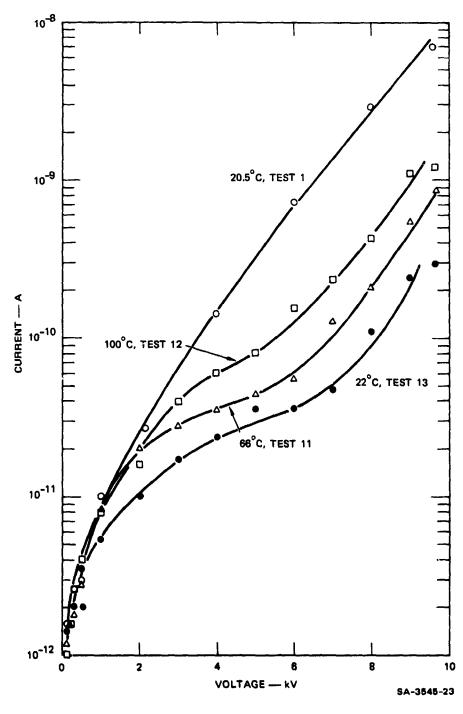


FIGURE 6 DARK, BULK CURRENT IN 0.0051-cm-THICK KAPTON-H BEFORE (Test 1) AND AFTER (Tests 11, 12, 13) ELECTRICALLY STRESSING AND HEATING IN A VACUUM

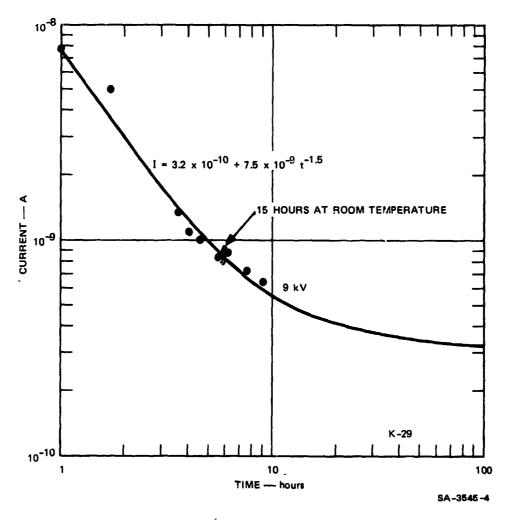


FIGURE 7 DARK, BULK CURRENT IN KAPTON 1.27 X 10⁻⁴ m THICK AT 100°C vs TIME IN VACUUM

great, it was found that if the samples were maintained at 100° C in a hard vacuum until the change in conductivity with time became inconsequential, the results from one sample to another were as predictable with Kapton-H as with any of the other materials tested.

These results were used to obtain the temperature, thickness, and electric-field dependences of the dark, bulk resistivity of Kapton shown in Figures 8 and 9.

All of the results discussed above were obtained from samples with metal electrodes. The measured dark, bulk current conducted through

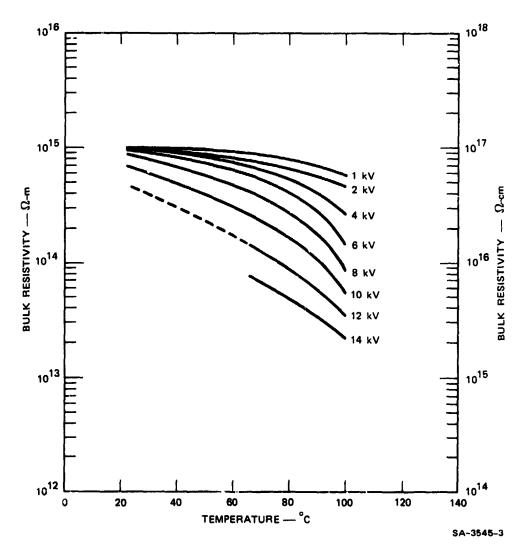


FIGURE 8 DARK, BULK RESISTIVITY OF KAPTON-H SAMPLE 0.0127 cm (5 mils) THICK vs TEMPERATURE AND APPLIED VOLTAGE

a Kapton sample 1.27×10^{-4} m (0.005 inch) thick immersed in an ionized-air plasma is shown in Figure 10. Also shown is a curve derived from several samples having metal electrodes. It is significant that measurements made with both positive and negative voltages applied to the electrode on the back of the sample resulted in currents of the same magnitude. It is also significant that the currents in these two types of experiments are so nearly equal in magnitude. In fact, the plasma electrode measurements were made on a sample that had been in the vacuum only a brief time

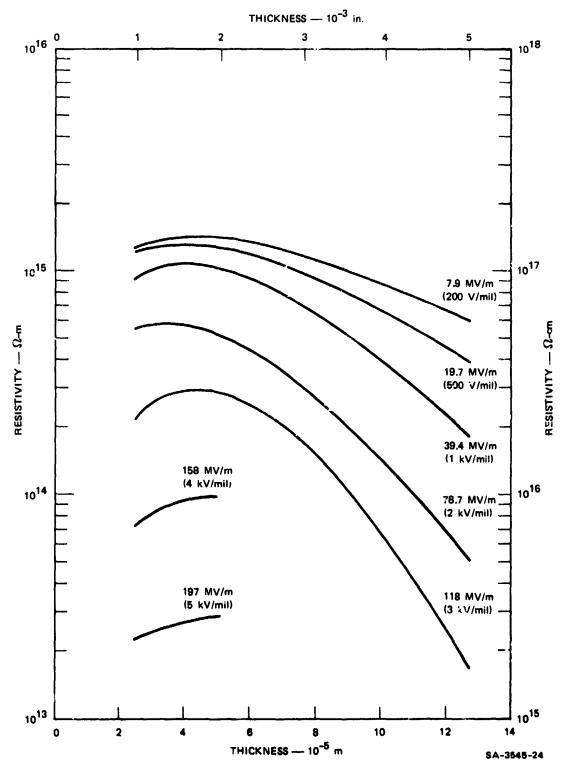


FIGURE 9 DARK, BULK RESISTIVITY vs THICKNESS AND ELECTRICAL STRESS AT 100°C — KAPTON

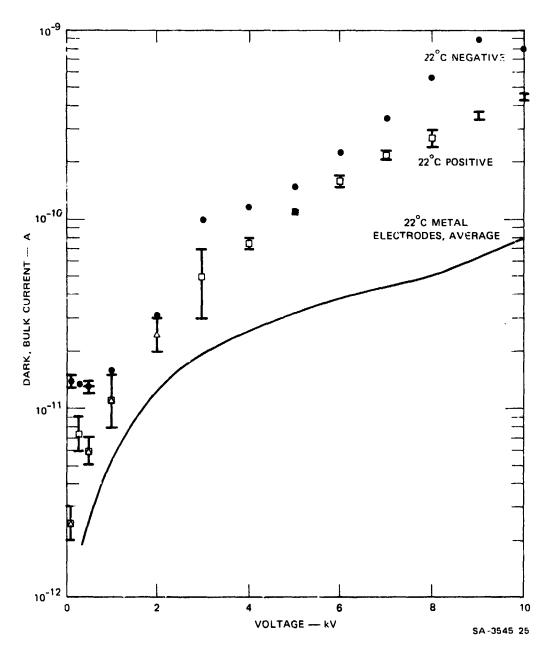


FIGURE 10 DARK, BULK CURRENT IN KAPTON 0.0127 cm (5 mils) THICK vs VOLTAGE FOR SAMPLES WITH METAL AND PLASMA ELECTRODES. Positive voltage attracts electrons and negative voltage attracts ions. Bars indicate variation of current during measurement.

and had not been elevated in temperature. Had this sample been aged more completely, it is consistent with all the earlier results (cf. Figure 7) to expect that the currents in the two types of experiments would have been nearly identical.

One of the major differences between measurements made with a plasma electrode and those made with metallic electrodes is in the rate at which the sample is charged. This rate is determined by the applied voltage and the circuit resistance. The circuit resistance is dominated by the 10^7 -ohm protection resistor in the case of metal electrodes, but can be dominated by the plasma resistance when a plasma electrode is used. The result of this higher effective resistance is that the charging time constant is increased quite severely. With metal electrodes, the current at t = 0 is V/R and equals 5×10^{-4} A at 5000 V. Since the sample capacitance is about 10^{-10} F, the charging time constant is about 10^{-3} s.

The contrast between the rapid charging when metal electrodes were used and the slow charging when a plasma electrode from which ions were extracted was used can be seen in Figure 11. The empirical fit shown indicates a time constant of 173 s--five orders of magnitude slower than with the metal electrodes. This result could be affected further by altering the RF power supplied to the multipactor and thereby changing the density of charge carriers in the plasma. It is clear that all these effects are artifices of the plasma and are not directly related to the material properties of the sample. Since the measurement interval of one minute used in tests with metal electrodes was not applicable in this case, other criteria had to be used in determining the current.

The current became reasonably stable in a period of 3 to 5 minutes when a positive potential was applied to the electrode and electrons were withdrawn from the plasma. This current was plotted in Figure 10. The bars on the data indicate the range over which the current varied in a period of 15 to 30 s. When the applied potential was

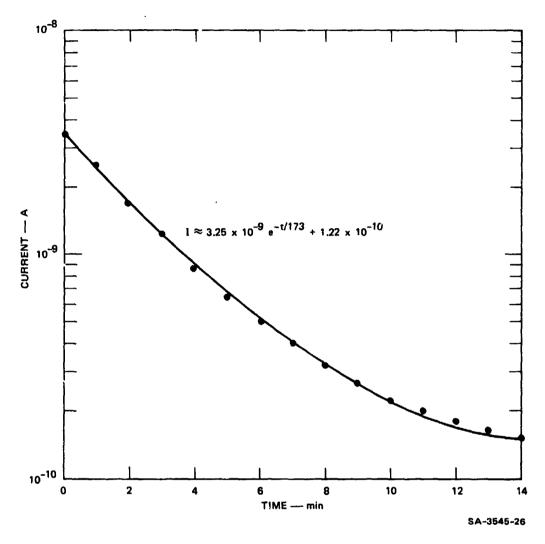


FIGURE 11 DARK, BULK CURRENT IN A KAPTON SAMPLE 0.0127 cm (5 mils) THICK USING A PLASMA ELECTRODE vs TIME. The applied voltage was -5000 V and ions were extracted from the plasma.

negative and ions were extracted from the plasma, the current reached equilibrium more slowly but the current was less erratic. Measurements were made in this case when the current was observed to decrease by less than 10% in a one-minute interval. Although these choices are somewhat arbitrary, the results indicate that the consistency is adequate.

Reiterating an earlier statement, the results of Figure 10 indicate that the conductivity of the sample is not dependent on the polarity with which the sample is charged, and is largely independent of whether metal or plasma electrodes are used. This result indicates that the dark testing of dielectrics for space use can be performed in several ways with the same results. The reasons for this will be discussed in Section IV.

2. Photoconductivity, Bulk

The greatest changes in conductivity observed in this program were those induced in Kapton by illumination with xenon light. Two important types of change were observed. The first of these was the change in conductivity during illumination, and the second was the change in the dark conductivity following illumination. This latter change, which appears to be permanent, led to considerable difficulty with the experimental procedures. For example, it was not possible to make photoconductivity measurements at two temperatures on the same sample because the illumination associated with the first measurements altered the electrical characteristics of the sample. Consequently, it was necessary to carefully choose the measurements to be made.

A particularly interesting set of measurements is shown in Figure 12. Measurements were made of the current conducted through a sample of Kapton 5.08×10^{-5} m (0.002-inch) thick held at 66° C while the sample was illuminated with light of varying intensity and spectral content. Measurements were made during and following a series of three-minute illuminations of the sample with a variety of filters. To remove any variations resulting from changes in the dark conductivity, the 3-kV potential was applied to the gold electrodes for a long period and no further changes in the dark conductivity could be observed. For reference, the dark current prior to the first illumination was 9×10^{-12} A. The measurements were begun in the red and IR, since the photons are least energetic in this range and the least change was anticipated. The test sequence and filter characteristics are given in the inset in Figure 12.

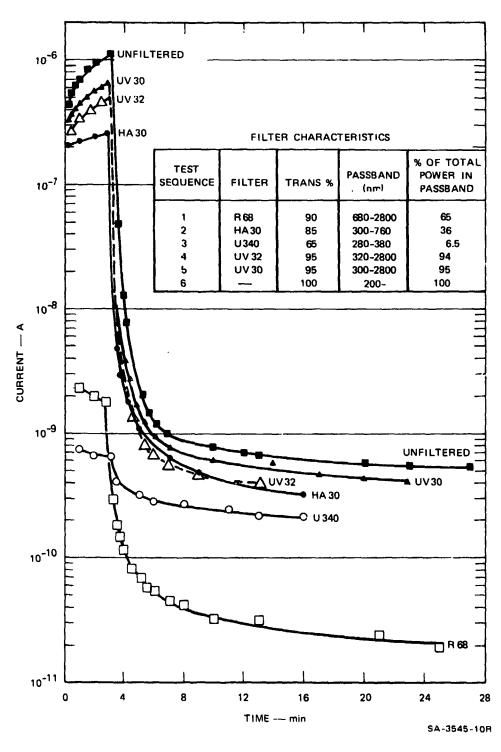


FIGURE 12 CURRENT DURING AND AFTER 3-MINUTE EXPOSURES OF KAPTON 5.08 \times 10⁻⁵ m THICK AT 66°C WITH VARIOUS FILTERS. Incident power on filters = 2.3 kW/m²; applied voltage = 3 kV; dark current prior to illumination = 9 \times 10⁻¹² A.

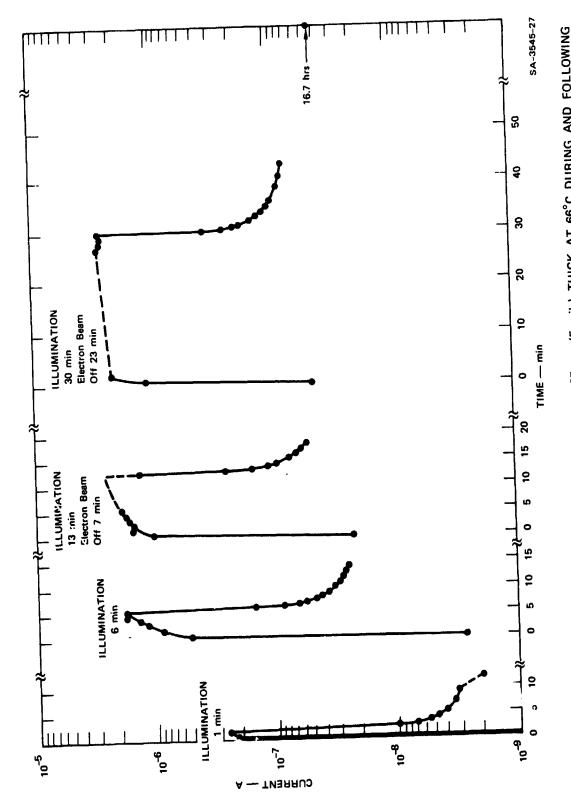
Illuminating the sample with light in the 680-to-2800-nm range (filter R-68) caused an increase in current from 9 \times 10⁻¹² to about 2 \times 10⁻⁹. Twenty-two minutes after the light was extinguished, the dark current was still twice the initial dark current.

The second test was performed with only half the total incident power of the test above, but resulted in a much greater increase in current to 2×10^{-7} A. This current is more than 20,000 times the initial dark current and the incident power is only 830 W/m² (1000 W/m² ~ 1 sun). The dark current 13 minutes after the light was extinguished was more than 30 times the initial dark current although the sample had been exposed to light for only six minutes.

The third test, in the 280-to-380-nm UV band, produced only a small photoconductivity. The incident power was only 1/6th of that in the preceding test, but the photoconductivity was reduced by a factor of 285. This indicates that the photoconductivity of Kapton in the UV is quite small and that the observed photoconductivity could be a result of imperfect cutoff of the filter outside the pass band. The sample appears to have continued to recover from the preceding irradiation during this test and the final dark current was lower than it was following the earlier test.

Subsequent irradiations with broadband or unfiltered light produced approximately equal incremental changes in the photoconductivity and remnant dark conductivity. As a result of these tests, entailing a total of 18 minutes of exposure of the sample, the dark conductivity increased by a factor of 61. Still larger changes were observed in another sample 65 hours after a series of illumination tests. The major photoresponse was induced by radiation in the 380-to-680-nm band.

The behavior using the electron gun as an electrode was very similar, as shown in Figures 13 and 14. Here the sample was exposed to



BULK CURRENT IN A KAPTON-H SAMPLE 0.0127 cm (5 mils) THICK AT 66°C DURING AND FOLLOWING ILLUMINATION. Electron-beam electrode; voltage applied to second-surface metal electrode = 5 kV. FIGURE 13

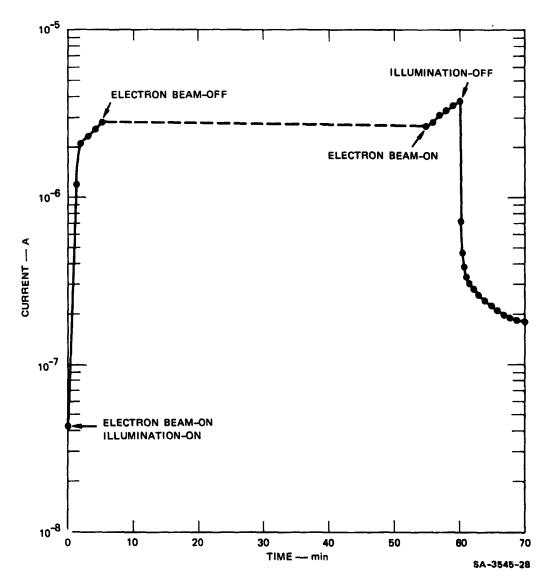


FIGURE 14 BULK CURRENT IN A KAPTON-H SAMPLE 0.0127 cm THICK AT 66°C DURING AND FOLLOWING ILLUMINATION. Electron-beam electrode; voltage applied to second-surface metal electrode = 5 kV.

illumination without filters for increasing periods of time. To avoid breakdown, however, the electron gun was turned off as indicated on the figures, but the voltage applied to the electrode remained on. During the period when the electron gun was turned off, the conductivity of the sample remained constant although the sample continued to be illuminated. This suggests that the change in photoconductivity is associated with the presence of electrons.

In an attempt at clarifying some of these phenomena, the optical densities of the various materials tested were measured as shown in Figure 15. Assuming a direct correlation between the optical density and the photocurrent, it is seen that the major photocurrent in Kapton should be stimulated by radiation of wavelength shorter than 600 nm. This measurement does not provide any additional information on the absence of a photocurrent with short-wavelength UV, however.

The complexity of the photoconduction process in Kapton was unexpected and is not fully understood at this time. It is clear that more experimental work on this material is in order.

3. Surface Resistivity

Surface resistivities can be strongly influenced by the condition of the sample surface. For this reason, samples of Kapton, as well as samples of other materials, exhibited surface resistivities that were somewhat variable. It was assumed that the highest resistivity observed corresponded to the true surface resistivity of the material and that lower resistivities were the result of surface contamination. The highest measured dark surface resistivities in Kapton exceeded 10¹⁷ ohms per square, although other samples were observed to have values lower by as much as a factor of 10². The higher value above approaches the limits of measurability of the system.

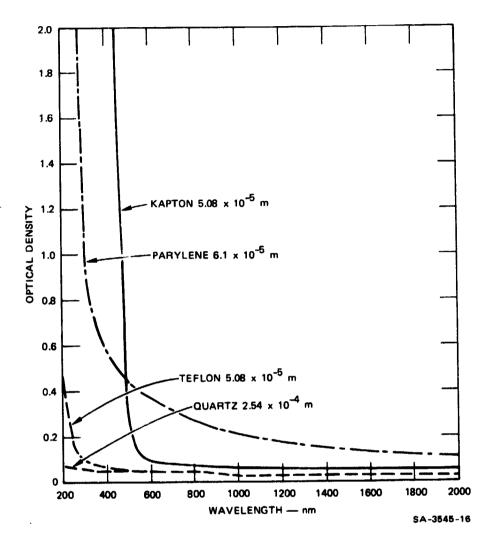


FIGURE 15 OPTICAL DENSITY OF TEFLON, PARYLENE, KAPTON, AND QUARTZ vs WAVELENGTH

Under illumination, the conventional concept of surface resistivity can become meaningless due to the photoemission of electrons from the negative electrode. These electrons are ejected from the cathode and freely move to the anode, masking any true changes in the surface properties. This situation could, in principle, be avoided by masking the electrodes to avoid exposure. Such a procedure was impractical in these experiments. Although the measured current is not associated with the surface per se, the current across the surface from this source can be appreciably greater than one would have anticipated on the basis of true surface resistivities.

B. Teflon

1. Dark Conductivity, Bulk

Although some minor changes in the dark, bulk conductivity of Teflon were observed after repeated testing in the vacuum, Teflon was found to be considerably more stable than Kapton. There were occasional samples in which the measured currents deviated substantially from those observed in other samples of the same thickness. This variation does not appear to be attributable to changes in the physical properties and is probably caused by small variations in the thickness of the samples. As with Kapton, the most consistent results were obtained with the thickest samples tested.

The dependence of the resistivity of Teflon on temperature is shown in Figure 16, with the applied voltage as a parameter. It is seen in the figure that the resistivity of Teflon is four to ten times greater than that of Kapton of the same thickness, and its variation with temperature is less rapid than it is in Kapton (cf. Figure 8).

The thickness dependence of the bulk, dark resistivity is shown in Figure 17. The thinnest samples had resistivities that were more temperature sensitive and sharply lower than in the thicker samples measured. Unlike Kapton, in which the resistivity decreased with increasing thickness, the resistivity of Teflon remains relatively constant in thicker samples.

2. Photoconductivity, Bulk

The photoconductivity of Teflon was dominated by UV effects. It was found that more than 90% of the photocurrent was caused by exposure of the sample to UV in the 200-to-300-nm band, as shown in Figure 18. In this figure, filter UV30 (cf. Figure 12) was used to exclude UV radiation. The change in conductivity caused by the filtered light is only slightly outside the scatter of the data obtained in the dark.

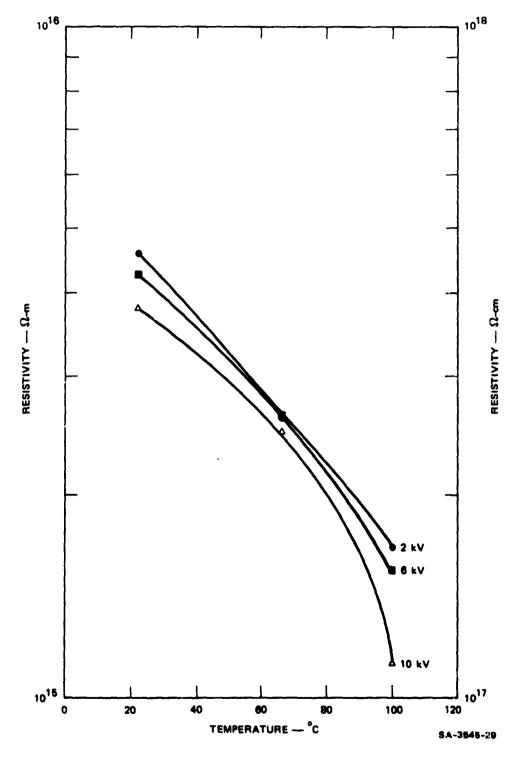


FIGURE 16 DARK, BULK RESISTIVITY vs TEMPERATURE AND VOLTAGE IN A TEFLON SAMPLE 0.0127 cm (5 mils) THICK

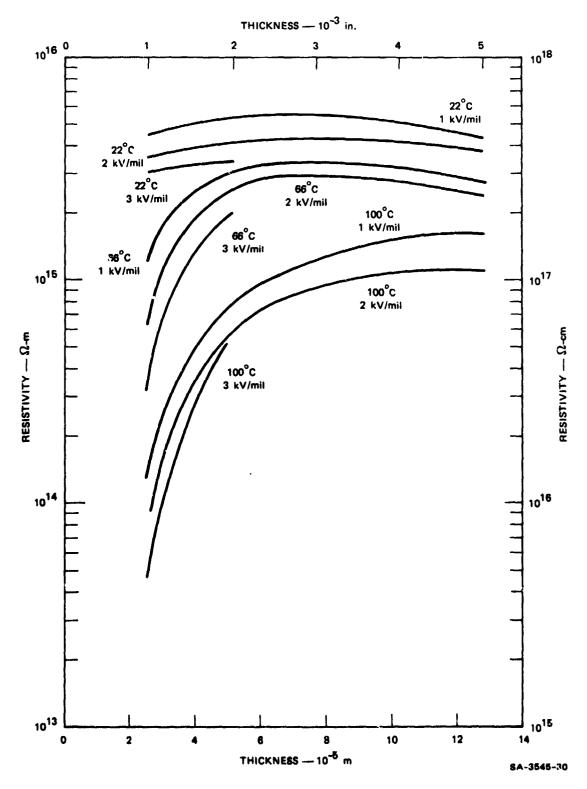


FIGURE 17 DARK, BULK RESISTIVITY OF TEFLON VS THICKNESS AND TEMPERATURE

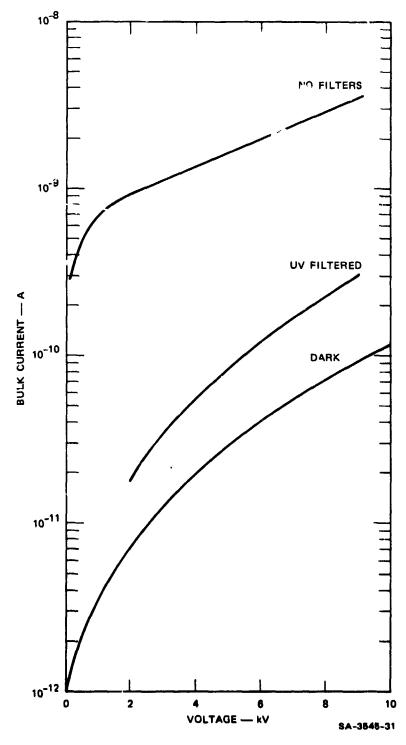


FIGURE 18 BULK, DARK CURRENT AND BULK PHOTOCURRENT IN A TEFLON SAMPLE 0.0127 cm (5 mils) THICK AT 100°C. Photocurrents measured with and without filtering of UV radiation having wavelengths shorter than 300 nm.

In an attempt at obtaining additional information on the photo-conductive behavior of Teflon, measurements were made of the photocurrent while exposing the sample to xenon light through a series of filters.

The normalized photocurrent is shown in Figure 19. In normalizing the data, the measured current was divided by the passband in nm and by the power incident on the sample. The illuminations used in these tests did not contain UV that would cause photoemission from the gold electrode (<280 nm)

Referring back to Figure 15, it is seen that, of the materials tested, only Teflon and quartz transmitted appreciable amounts of light in the UV. It will be shown later that the major photocurrent in quartz is also caused by irradiation in the UV. Although definitive experiments were not performed, it is believed that the UV-induced photocurrent is a result of photoemission of electrons from the back (negative) gold electrode on the sample and is not an intrinsic property of the material tested. The intrinsic photoconductivity was most responsive to illumination

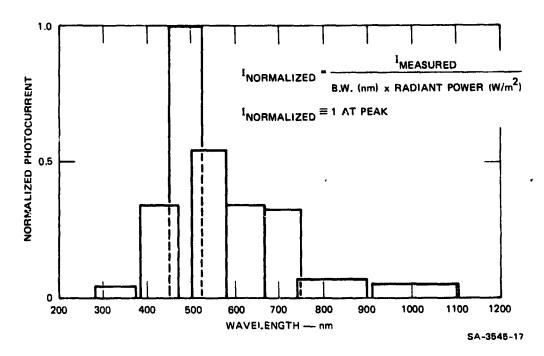


FIGURE 19 NORMALIZED PHOTOCURRENT vs WAVELENGTH — TEFLON, 100°C, 39.4 MV/m. Tests did not extend far enough into UV to stimulate photoemission from electrode.

in the 400-to-750-nm band, and peaked around 500 nm. No permanent changes in the conduction properties of Teflon were noted after exposure to xenon light.

3. Surface Resistivity

The highest measured dark, surface resistivity in Teflon exceeded 10^{17} ohms per square, although one sample was measured that had a value lower by a factor of 10^4 . It is possible that this latter value was influenced by gold that diffused under the mask during deposition. The surface resistivity in the light was dominated by photoemission, as was the case with Kapton.

C. Quartz

1. Dark Conductivity, Bulk

Measurements were made of the dark, bulk current through samples of clear, bubble-free fused quartz having no visible fluorescence under illumination with xenon light. The samples were in the form of circular discs 2.54×10^{-4} m (10 mils) thick and 0.102 m (4 inches) in diameter. Measurements were made at 22, 66, and 100° C. The measured currents were quite small, and several sets of data were averaged to obtain the resistivities shown in Figure 20. The currents measured at 22° C were below 10^{-12} A at most voltages and were not sufficiently reliable to define the resistivity-versus-voltage curve.

2. Photoconductivity, Bulk

As with Teflon, the current conducted through the quartz sample during illumination was largely governed by the UV content of the light. The photocurrent was reduced by about a factor of 10 when the UV light having wavelengths shorter than 300 nm was blocked (Figures 21 and 22).

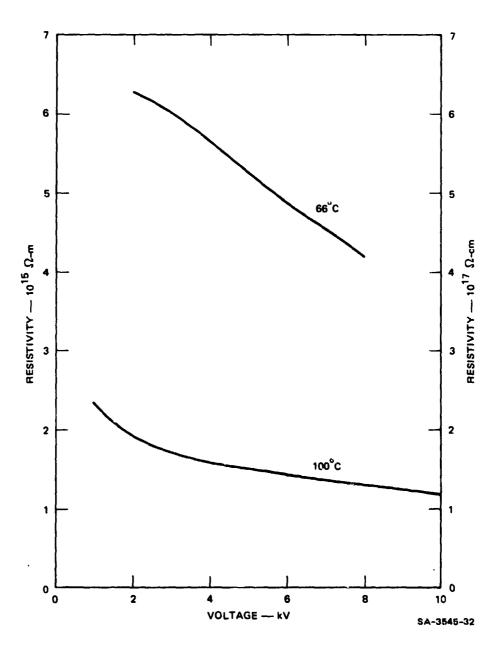


FIGURE 20 DARK, BULK RESISTIVITY IN A FUSED QUARTZ SAMPLE 0.0254 cm (10 mils) THICK vs VOLTAGE AND TEMPERATURE

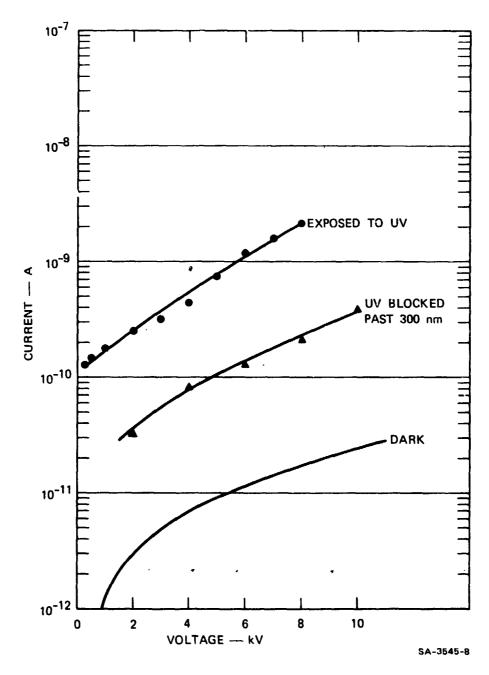


FIGURE 21 BULK, DARK CURRENT AND BULK PHOTOCURRENT IN 10-mil-THICK FUSED QUARTZ AT 100°C vs VOLTAGE WITH AND WITHOUT UV BLOCKING BEYOND 300 nm. Light intensity = 2.5 kW/m².

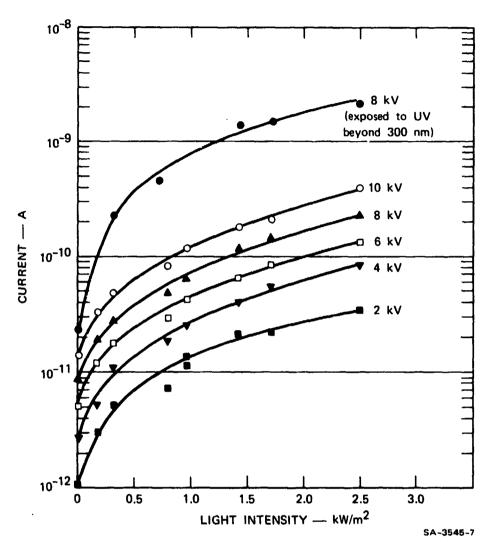


FIGURE 22 BULK PHOTOCURRENT IN 10-mil-THICK FUSED QUARTZ AT 100°C vs LIGHT INTENSITY WITH AND WITHOUT UV BLOCKING BEYOND 300 nm

Since most of the UV is transmitted through the sample (see Figure 15), and since photoemission in gold occurs in the 300-to-260-nm (4.2-to-4.8 eV) spectral band, it is reasonable to believe that the large UV dependence of the photocurrent is caused by electrons released by the rear (negative) gold electrode. This appears to be the dominant source of photoconduction, and the residual current could be a result of incomplete blocking of light in this spectral band by the filter.

3. Surface Resistivity

Surface photocurrents were measured with and without UV having wavelengths shorter than 300 nm. As is usually the case in such measurements, the magnitude of the surface current depended on the condition of the surface. The highest surface currents occurring with the UV blocked are shown in the lower curve of Figure 23. These data were obtained after the sample remained at a pressure of about 1 torr overnight. The current fell by about a factor of three when the sample was heated in a hard vacuum and was approximately the same as the dark surface current. The surface resistivity at 10 kV with the UV blocked was $2.3 \times 10^{16} \Omega/\Box$; values in excess of $7 \times 10^{16} \Omega/\Box$ were measured under cleaner conditions.

The true surface current of the material cannot be determined when the incide t radiation contains UV in the 200-to-300-nm range due to photoemission from the gold guard electrode. These electrons are emitted from the guard ring at ground potential and are attracted to the center, positive electrode and appear as a surface current. The measured current, shown in the upper curve of Figure 23, is typical of the results in this case.

D. Parylene

1. Dark Conductivity, Bulk

The dark conductivity of Parylene was most notable for its temperature stability over the range of temperatures studied. The resistivity was virtually unchanged from 22 to 66° C, and increased by less than a factor of three at 100° C.

Although the resistivity of this material was quite stable with temperature, it was also lower than in any of the other materials studied. Only one sample of Parylene 6.1 \times 10⁻⁵ m (2.4 mils) thick was tested and the voltages were kept to modest values to avoid dielectric

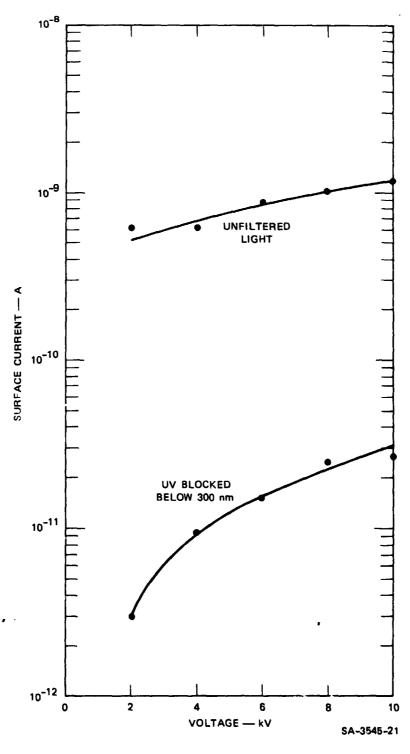


FIGURE 23 SURFACE PHOTOCURRENTS IN QUARTZ 0.0254 cm (10 mils) THICK vs VOLTAGE AT 66°C, SHOWING EFFECT OF UV RADIATION ON SURFACE CONDUCTIVITY

breakdown. Over the temperature range from 22 to 100° C and with voltages from 100 to 4000 V, the resistivity varied from 2.6 × 10^{13} to 10^{14} Ω -m.

2. Photoconductivity, Bulk

The photoconductive properties of Parylene were also remarkable for their stability. Under 2.5 kW/m 2 of illumination, the conductivity increased only by a factor of approximately five, and was comparable to quartz and Teflon under the same conditions. With the UV blocked, the photocurrent was almost independent of temperature over the range tested.

The photoconductivity of Parylene was smaller and less dependent on the UV content of the illumination than was the case with quartz and Teflon. If the UV-enhanced photoconductivity in the latter two materials is caused by photoelectrons emitted from the rear electrode as supposed, then the absence of a UV-enhanced photocurrent in Parylene can be explained by its opaqueness in the UV (see Figure 15). The absorption of the UV by the specimen prevents the emission of electrons by the rear electrode.

The photocurrent in Parylene, normalized to remove spectral variations in the output of the source and the bandwidth of the filters, is shown in Figure 24. By comparing this figure with Figure 19, it can be seen that the photoresponse of Parylene extends over a wider spectrum than does the photoresponse of Teflon.

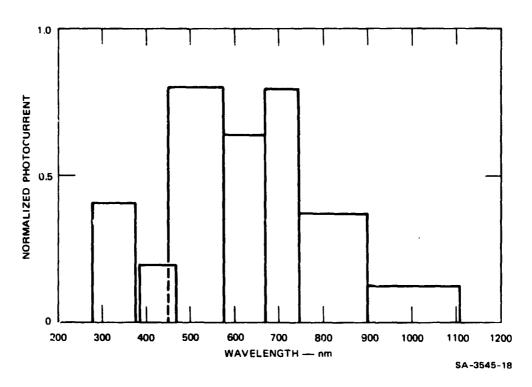


FIGURE 24 NORMALIZED PHOTOCURRENT vs WAVELENGTH — PARYLENE, 22°C, 32.8 MV/m

IV DISCUSSION OF RESULTS

A. General

Crystalline, inorganic insulators, are generally analyzed as semiconductors having large energy gaps between the valence and conduction
bands. Since the gap is large, few electron are available for conduction
at ordinary temperatures and the resistivity is very high. Considerable
effort has been devoted in the past to establishing the lattice and conduction properties of crystalline samples of semiconductors and insulators. The study of the conductivity of polymeric insulating materials,
however, is a comparatively infant discipline and is much more poorly
understood.

It is possible to have several conduction phenomena occurring simultaneously in a sample, each with its own characteristic behavior. Conduction can be intrinsic, in which case electrons are transferred from the valence to the conduction band, or extrinsic, in which case impurities in the host material contribute the charge carriers. Charge carriers can also arise from the electrodes attached to the sample, and annihilation of carriers can occur at the surface depending on the nature of the ambient atmosphere. Charge carrier traps can affect the voltage and temperature dependence of the conduction current and can have a profound effect on the photoconductivity of a sample.

Photoconduction generally occurs when electrons are excited from the valence band to the conduction band by absorption of an incident photon. Similar effects can occur when the photon's energy is absorbed by an electron, hole, or ion trapped at an imperfection in the lattice. The latter mechanism leads to an enhanced conductivity after the

illumination is extinguished, since some time is required before the charge carriers again become trapped at an imperfection. This recombination time can be very short or very long--in some cases, weeks. It was impractical, in the experiments discussed herein, to observe these long recombination times although some short-time measurements were recorded.

B. Dark, Bulk Resistivity

The measured dark, bulk resistivities of the four materials studied are compiled as functions of the applied electric field in Figures 25, 26, and 27 at temperatures of 22°C, 66°C, and 100°C, respectively. The Kapton data shown are those acquired after stabilization of the samples.

The most prominent feature of these data is the sharp decrease in resistivity (or increase in conductivity) in intense electric fields. The only exception to this behavior was noted with the thinnest Teflon samples.

The data for the Kapton sample 1.27×10^{-4} m thick at 100° C are replotted in Figure 28 on a Schottky plot, where it is seen that at high fields the data are well fitted by an $\exp(aE^{1/2})$ function. This function describes the electric-field-enhanced thermionic emission of electrons from the negative electrode into the dielectric. The theory assumes that the electrons enter the conduction band and are immediately available in the conduction process. The data obtained at 66° C in the same sample indicate similar behavior at high fields, as do the data for the thinner Kapton samples at 100° C. An inspection of Figures 25 through 27 shows that a similar behavior occurred in Teflon and Parylene. At lower fields and at lower temperatures, however, conduction is clearly due to different processes.

In all cases the resistivity is expected to decrease as the temperature is increased. This is a result of the increased number of electrons

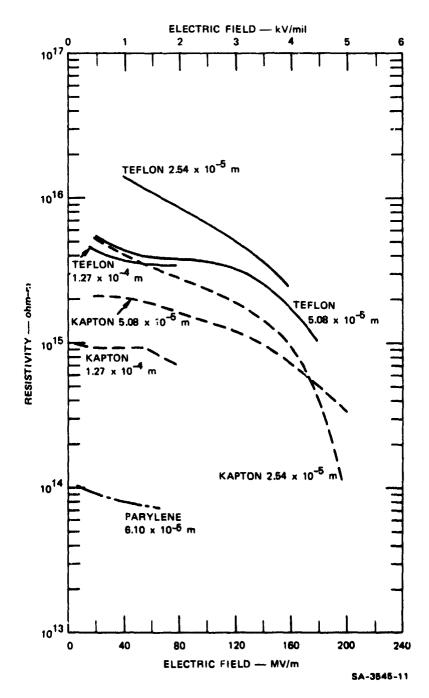


FIGURE 25 DARK, BULK RESISTIVITIES OF INSULATORS VS ELECTRIC FIELD AT 22°C

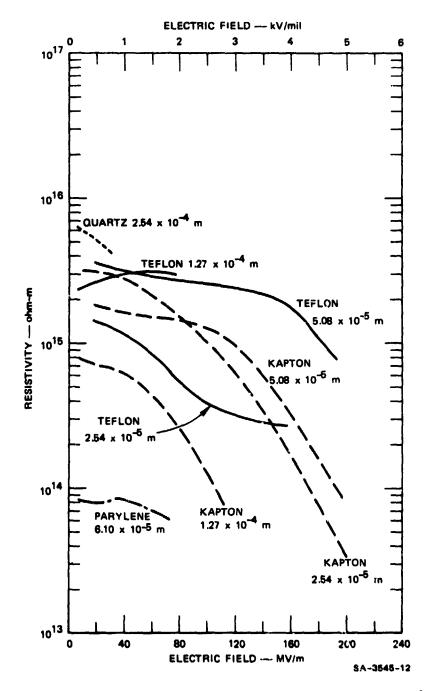


FIGURE 26 DARK, BULK RESISTIVITIES VS ELECTRIC FIELD AT 66°C

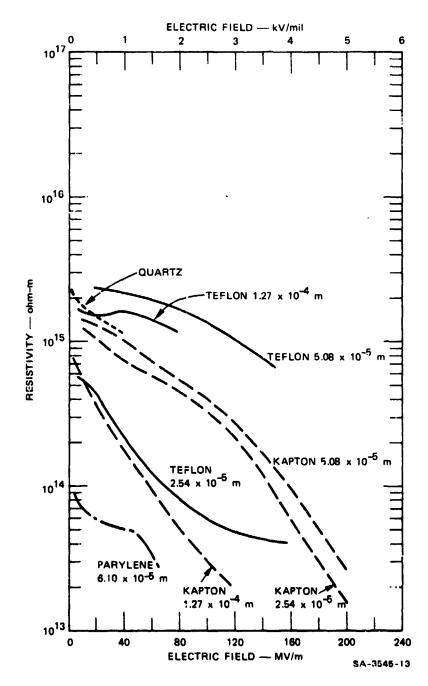


FIGURE 27 DARK, BULK RESISTIVITIES OF INSULATORS VS ELECTRIC FIELD AT 100°C

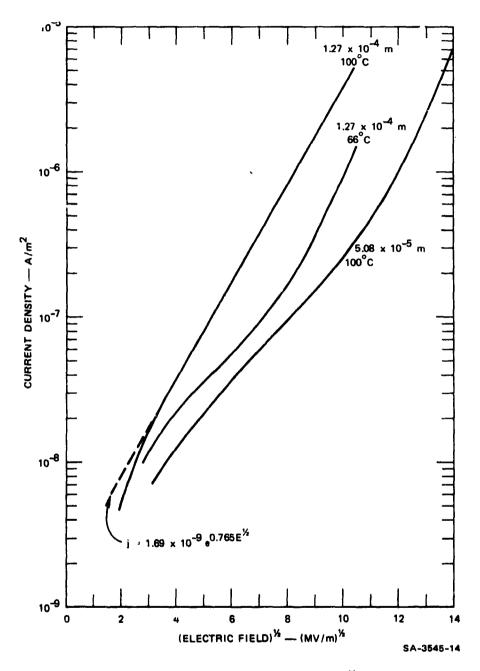


FIGURE 28 CURRENT DENSITY vs (Electric Field) 1/2 - KAPTON

in the conduction band and the increased mobility of the charge carriers. This characteristic was observed to be the case with all the materials tested including Kapton after it was stabilized.

Prior to the stabilization of Kapton the current density was proportional to the hyperbolic sine of the applied voltage. According to the theory of ionic conduction,

$$j = \frac{\sigma_0 V}{t} \exp(-\theta/k_0 T) \sinh \left(\frac{e^{dV}}{2k_0} Tt\right)$$

where t is the sample thickness, Ø is an activation energy, e is the electron charge, k is Boltzmann's constant, V is the voltage, T is the absolute temperature, and d is the distance an ion "hops" each time it changes positions. The curves fitted to the data in Figure 5 yield hopping distances of 13 to 24 Å. increasing with the thickness of the sample. These results are comparable to those found for polyethylene.²

It was shown in Figure 6 that the shapes of the j-versus-V curves were changed soon after the sample was placed in a vacuum. This change was accelerated by heating the sample and was affected further by the application of a voltage. The variation with time at constant temperature and voltage was shown in Figure 7. These data suggest that the virgin conductivity of Kapton is dominated by the conduction of adsorbed ions. The ions are made more mobile by heating and by applying an electric field, and are soon removed from the conduction process by migration to the surface where they are trapped or evaporate. Parmley et al. have measured the outgassing of Kapton and found that water vapor is evolved (together with other gasses) under similar circumstances. It therefore is reasonable to believe that the initial conductivity in Kapton is caused by adsorbed

 $^{^{\}star}$ References are listed at the end of the report.

water. After evaporation of this water, the conductivity at high fields is dominated by field emission from the electrodes. This latter behavior is also found in the other materials investigated.

The electrical conduction through Kapton was investigated using both metal (gold) electrodes and free-charge electrodes, the latter being comprised of the ionized residual gas in the vacuum chamber. The results, previously presented in Figure 10, indicated that the conductivity was of the same order of magnitude with both types of electrode. The resistance of the plasma was measured and found to be of the order of 10^8 ohms when electrons were extracted from the plasma, and 10^{11} ohms when ions were extracted. Typically, the resistances of the samples were in the range of 10^{12} to 10^{15} ohms. For this reason, most of the potential drop occurs across the sample itself rather than across the plasma, so the plasma can be regarded as a good electrode for these experiments.

Although the nonmetallic electrodes had little effect on the ultimate result, the time required to establish equilibrium was radically altered. If the resistance of the circuit external to the sample is taken to be R, and if the resistance of the sample is taken to be R_s , and its capacitance to be C, then the current at time t after applying a voltage V can be shown to be:

$$i = \frac{V}{R + R_s} \left[1 + \frac{R_s}{R} \exp \left(-\frac{R + R_s}{RR_sC} t \right) \right]$$

In these experiments, R $_{\rm S}$ was always greater than R, and the current can be approximated by

$$i = \frac{V}{R_s} + \frac{V}{R} e^{-t/RC}$$

where the first term is the equilibrium leakage current. Unless the resistance external to the sample is comparable to the sample resistance, this equilibrium current is independent of the external resistance. An expression of this form was used to fit the data of Figure 11.

The second term is the charging current and varied over a wide range, depending on the experimental conditions. With direct electrical contact, as in the metal electrode experiments, R was controlled by the 10^7 -ohm protection resistance in the circuit. This external resistance was dominated by the plasma resistance when nonmetallic electrodes were used. Consequently, the charging current could be much lower and the charging time constant much longer when the plasma electrode was used, although the equilibrium current would be the same whether the plasma electrode or a metallic electrode was used. Fitting the above expression for the current to the data of Figure 11 results in a sample resistance of 4.1×10^{13} ohms, an external resistance (primarily plasma) of 1.5×10^{12} ohms, and a capacitance of 1.1×10^{-10} farads.

The maximum charging current of 3.3×10^{-9} A occurs at t=0. If R were 10^7 ohms as in the metal-electrode experiments, the maximum charging current would have been 5×10^{-4} A at t=0, and the time constant would have been 1.1×10^{-3} s. If the measurements of the current had been made at 60 s in both cases, the equilibrium current would have been in error by a substantial margin. The current in the metal-electrode experiments would have reached equilibrium at 1.22×10^{-10} A, whereas the current with the plasma electrodes would still be dominated by the charging current at 2.4×10^{-9} A. Because the charging time constant is long when the plasma electrode is used, small changes in the potential applied to the sample result in charging currents that persist for a prolonged period. This feature requires that a highly regulated power supply be used in the experiments.

The comparable magnitudes of the current when positive and negative potentials were applied to the second-surface electrode were noted earlier. This is the result that would be expected if the mobility of ions were very low in the sample. In this case, electrons extracted from the plasma would be conducted through the sample if the electrode were positive. However, if the electrode were negative, ions would be extracted from the plasma and establish a potential across the sample. Electrons emitted from the gold electrode would then be conducted through the sample and recombine with the ions to complete the charge transfer. The mobility would then be determined by the mobility of the electrons in either case.

C. Surface Conductivities

Surface conductivities in the dark were approximately 10¹⁷ ohms/
in all materials in the best cases. It is assumed that the lower resirtivities noted in some cases were caused by surface contamination of the samples. The surface conductivity in the presence of an electron gas or an air plasma was not measured because the plasma would effectively short-circuit the electrodes. The surface conductivity in the presence of a plasma could not reasonably be measured, first-surface electrodes were not applied to the samples used in these tests. Similarly, surface conductivities in the presence of xenon light were dominated by the conduction of electrons photoemitted from the negative to the positive electrode.

D. Bulk Photoconductivity

Although the bulk photoconductivity characteristics of Teflon and quartz were similar, the characteristics of Parylene and Kapton-H differed significantly from each other and from quartz and Teflon.

The bulk photoconductivity of Teflon and quartz increased by a factor of ten or less when exposed to 2.5 kW/m 2 of xenon illumination if UV

radiation of wavelength shorter than 300 nm was blocked. When exposed to short wavelength UV, however, the photocurrent in both materials increased by an additional factor of approximately ten (cf. Figures 18, 21, and 29). This result suggests that the strong photocurrent in the UV is caused by the photoemission of electrons from the gold electrodes on the second surfaces of the samples. For this assumption to be valid, the UV radiation must reach the second surface--i.e., the material under test must transmit UV without excessive absorption. Measurements of the optical density of quartz and Teflon indicate that the absorption of UV in these materials is low (cf. Figure 15). Both materials were relatively good insulators in the absence of short-wavelength UV, as shown in Figure 29.

Parylene exhibited less change in conductivity under illumination than any of the other materials tested. Although this material was a comparatively poor insulator in the dark, it was comparable with Teflon and quartz when each of these materials was illuminated with light containing short-wavelength UV. Unlike quartz and Teflon, Parylene does not transmit any appreciable amount of short-wavelength UV (cf. Figure 15) and photoemission from the gold electrode is not expected to affect the photoconductivity to any great extent.

The photoconductivity of Kapton was notable for its great magnitude and the persistence of the increased conductivity after the light was extinguished. The bulk conductivity in the presence of 2.3 kW/r of illumination was as much as 5 orders of magnitude higher than the bulk conductivity in the dark prior to the illumination (cf. Figure 12). The resistivity of Kapton under this illumination was 4.2 x 10 ohm-meters, three orders of magnitude lower than in any of the other materials tested. A long recombination time for the charge carriers is characteristic of very good photoconductors. It appears, however, that the illuminations of Kapton might have caused the conductivity to be permanently increased-

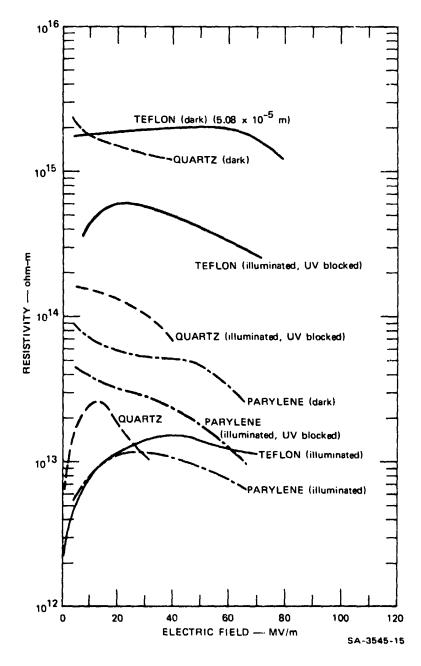


FIGURE 29 DARK, BULK RESISTIVITIES AND PHOTORESISTIVITIES OF TEFLON, QUARTZ, AND PARYLENE WITH AND WITHOUT UV RADIATION — 100°C

possibly caused by a photochemical change in the polymer. It will be necessary to make measurements over a longer period of time than was possible in this program to establish whether the observed changes were permanent.

The data shown in Figures 13 and 14 illustrate another interesting feature of the photoconductivity of Kapton. These measurements, made with an electron-beam electrode, indicate that the conductivity of Kapton increased under illumination only so long as the electron beam was applied to the samples. This result suggests that the charge carriers in the photoconductive process are electrons that were freely supplied by the gold electrode when these were used, but were supplied by the electron beam in the later experiments. If this is the case, the observed changes in the dark conductivity are the result of untrapped charge carriers in the materials. That this condition should persist for such long periods of time is unusual and should be investigated further.

V CONCLUSIONS

Many diverse phenomena were observed in this program that can be of importance to the spacecraft designer. Although some of these phenomena may lead to poor insulation in the traditional sense, it may be possible to use these phenomena in solving present or future problems in the design of spacecraft--such as the problem of spacecraft charging.

In the dark, FEP Teflon, Kapton-H, and quartz were found to be good insulating materials, each having bulk resistivities approaching or exceeding 10 ohm-meters. These values were attained in Kapton-H, however, only after the samples had been electrically stressed and heated in vacuo for several hours. Parylene was found to be a relatively poor dark insulator. Both Parylene and Kapton-H exhibited elecret effects both in the bulk and surface resistivity measurements. As with the bulk resistivity, Parylene, FEP Teflon, Kapton-H, and quartz were good surface insulators in the dark, all having surface resistivities of the order of 10 ohms/ in clean samples. The dark resistivity of Kapton-H was significantly decreased after exposure to xenon light. Although some recovery was observed, changes in the dark conductivity as great as a factor of 10 were observed 65 hours after the light was extinguished.

The photoconductive properties of quartz and Teflon were dominated by effects related to the incidence of short-wavelength UV. This was true both in the bulk photoconductivity, where photoelectrons were emitted from the negative gold electrode and greatly increased the conductivity through the sample, and in the surface conductivity, where a similar effect occurred. Both materials were found to be relatively transparent to UV capable of causing photoemission from gold. When the UV was blocked, the conductivity of both quartz and Teflon decreased by about an order of

magnitude. The photocurrent in Teflon was most responsive in the vicinity of 500 nm and dropped rapidly below 400 nm and above 750 nm.

The photoconductivity of Parylene was comparatively stable at a relatively high value with or without UV radiation. This effect is tentatively ascribed to the absorption of UV in the bulk of the specimen, preventing photoemission at the cathode. The photocurrent in this material was found to respond over a wider spectral band than in Teflon. Appreciable currents were induced by radiation from 280 to 900 nm in wavelength.

Photocurrents in Kapton-H were induced primarily by radiation having wavelengths from 380 to 680 nm. This response was relatively intense; the conductivity varies by as much as five orders of magnitude between the dark and 2.5 kW/m ² of illumination. The resistivity under this illumination was 2 to 3 orders of magnitude lower than in any of the other materials tested in the presence of UV radiation. To achieve this pronounced increase in conductivity, it was necessary that electrons be present--i.e., illuminating the sample in the absence of a metal electrode or an electron beam did not cause an increase in conductivity.

Plasma electrodes of either positive or negative polarity or an electron-beam electrode resulted in the same ultimate steady-state current as metal electrodes. The greatly increased resistivity of the plasma electrode, however, increased the charging time by as much as five orders of magnitude.

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